SANDIA NATIONAL LABORATORIES
CIVILIAN RADIOACTIVE WASTE MANAGEMENT
TECHNICAL PROCEDURE

TP-202

Revision 03

Measurement of Thermal Conductivity of Geologic Specimens Using the Guarded Heat-Flow Meter Method

Effective Date: 10/15/01

Original signed by Nancy S. Brodsky	*10/5/01*
Author: Nancy S. Brodsky	Date
Original signed by David R. Bronowski	*10/5/01*
Technical Reviewer: David R. Bronowski	Date
Original signed by James F. Graff	*10/8/01*
Quality Assurance Reviewer: James F. Graff	Date

(Reviewer signatures above serve to document the review and resolution of comments.)

REVISION HISTORY

Revision	Description
00	Initial Issue
01	Full revision to address QAIP 20-1 requirements, make other minor improvements, and incorporate change in location of equipment from Holometrix Inc. to SNL
02	Revised to incorporate new moisture containment cell design and to permit the use of quartz standards for calibration.
03	TP 202 was deactivated during Audit BSC-ARC-01-010. It is now reactivated for the work to be performed under TWP *Subsurface Performance Testing for License Application (LA) for Fiscal Year 2001, TWP-EBS-MD-000009 Rev. 03*. No major technical revisions were required from revision 2, only reference to current procedures have been revised (i. e., WAs to TWPs, QAIPs to APs) and other minor editorial revisions.

TABLE OF CONTENTS

1.0 SCOPE AND OBJECTIVE

This Technical Procedure (TP) applies to the measurement of thermal conductivity using a guarded heat-flow meter. This TP is specifically intended for implementation in the Sandia National Laboratories (SNL) Yucca Mountain Site Characterization Project. The basic method is similar to that described in the American Society for Testing and Materials (ASTM) procedure for evaluating the thermal conductivity of gasket materials (F433-77). The objective of the activity described in this TP is to measure the thermal conductivities of geologic materials over a temperature range of approximately 30 °C to 200 °C.

Thermal conductivity (K_{th}) of a material is defined as the time rate of steady state heat flow through a unit area per unit temperature gradient in the direction perpendicular to an isothermal surface. Thermal conductivity is expressed in W/mK.

2.0 PREREQUISITES

Before performing this technical procedure, personnel must be trained by the Principal Investigator and demonstrate their proficiency in performing this procedure. The Principal Investigator has responsibility for generating a record of personnel proficiency training.

Personnel using this technical procedure are responsible for ensuring that a current controlled copy of this procedure is available and used for performing this procedure.

3.0 ACTIVITY DESCRIPTION

The guarded-heat-flow-meter method is one of several techniques for measuring thermal conductivity of geologic specimens. In general, the guarded heat-flow meter can be used to measure thermal conductivities between 0.1 and 10 W/mK.

With the guarded-heat-flow-meter method, a specimen is placed between two heater plates at different temperatures, resulting in heat flow through the specimen. The heat flow is measured by a thin heat-flux transducer (HFT) located between the specimen and one of the heater plates. A cylindrical guard heater around the specimen, maintained at near the mean specimen temperature, reduces radial heat flow losses. Since the guard heater is set at a mean temperature, rather than matching the thermal gradient in the specimen, shorter specimen lengths (generally less than 20 mm) are required for this method than for other techniques. Slightly larger specimens can be used if the machine output remains linear with increasing specimen lengths.

At thermal equilibrium, the specimen thermal resistance R_S (m^2 °K/W) is determined from the equation

$$R_s = \frac{N\Delta T}{Q} - R_0$$

where N is the constant of proportionality between the HFT output and the heat flux in $m^2\mu V/W$, ΔT is the temperature difference between the upper and lower faces of the specimen in degrees Kelvin, Q is the HFT output in μV, and R_O (in m^2K/W) is the offset resulting from a combination of residual thermal resistance at the contacts between the specimen and the heater plates and nonlinearity in the HFT output at low output voltages. The constants N and R_O are determined by calibrating the heat-flow meter with reference specimens of known thermal resistance.

The thermal conductivity of the specimen, $K_{th,}$ (W/mK) is obtained from

$$K_{th} = d/R_S$$

where d is the specimen thickness in meters. The specimen thickness is determined in accordance with TP-200, *Inspection of Samples Used In Thermal Properties Measurements*; it is measured to an accuracy of ± 0.05 mm.

4.0 APPARATUS

The guarded heat-flow meter that will be used in following this TP is the C-Matic Model TCHM-LT (LT) manufactured by Holometrix Inc. This unit can be operated over the temperature range 0 °C to 200 °C.

To perform thermal conductivity measurements on saturated specimens, the specimen is placed in a moisture containment cell (see Figure 1A: 1.5 inch Diameter Moisture Containment Cell; 1B: 2.0 inch Diameter Moisture Containment Cell). Thermal conductivities of saturated specimens are only measured at temperatures below boiling (100 °C).

Precision statements for the LT have been determined according to procedures described in ASTM E177-86. Measurements on the LT are found to be repeatable (95% repeatability limit) to within ±3%. The instrument is calibrated and verified using reference materials that qualify as national standards (determined in accordance with AP-12.1Q, *Control of Measuring and Test Equipment and Calibration Standards*). The major source of uncertainty in measurements is the uncertainty in the thermal conductivity values of the reference materials. In this TP, high-purity (99.99%) clear fused quartz and Pyrex 7740 specimens are used for calibration and verification. Recommended thermal conductivity values for these materials have been published by the National Bureau of Standards in NSRDS-NBS-8 (Powell et al., 1966). For the temperatures attained in following this TP, uncertainties of ±5% (~0.07 W/mK) in the recommended values are cited for both Pyrex and high-purity fused quartz. Thermal conductivity measurements made in accordance with this TP are therefore considered accurate to within this limit. In addition to these materials, a verification may be performed using a specimen of Vespel SP1. Although this is not a NIST-traceable material, the manufacturer of the LT has accumulated a substantial amount of data on this material and uses it as a calibration standard. It is only used here as an additional verification standard.

4.1 Instruments

A diagram of the LT guarded heat-flow meter is shown in Figure 2. A specimen is placed above the HFT, and a pneumatic piston lowers the upper heater on to the specimen with a reproducible load. Type-K thermocouples are used to measure the temperatures of the guard heater (T_g), the control heater (T_h), and both specimen faces (T_u and T_1). The diagram indicates the thermocouple locations.

Thermocouples are calibrated by a Calibration Laboratory in accordance with AP-12.1Q, *Control of Measuring and Test Equipment and Calibration Standards*. The HFT (which gives an output, Q, proportional to the heat flux) is calibrated in accordance with Section 5.2.2.1. The loading pressure is monitored by a pressure gauge on the regulator which is mounted to the gas cylinder; ***this gauge is for indication only***.

4.2 Data Acquisition

4.2.1 Hardware

The computer hardware for the LT includes an IBM-compatible PC, a Lawson Labs 140 A/D board (for calibration, see TP-215, *Calibration of Lawson Board Systems)*, at least one disk drive for a portable disk, a keyboard, printer, and a monitor. The LT is equipped with a Lawson Labs 20B thermocouple board. A constant temperature-circulating bath is required for tests in which the temperature is less than 50 °C.

4.2.2 Test Control and Data Acquisition System

The test control and data acquisition system for the LT consists of "TCA200LT.EXE". .

Calibration data are given unique file names. A calibration data file name consists of 8 characters in the format "ABYYMMDD" where "AB" is either the operator's two initials or another identifier, "YY" is the last two digits of the year, "MM" is the month, and "DD" is the day (the date the calibration was initiated) followed by a 3 digit extension ".LTC". Raw data files are also given unique file names. A typical calibration or verification raw data file name consists of 8 characters in the format “ABYYMMDD” just as above, however, the file would have “.RAW” as the extension. An alternative to this can occur when several calibration files are generated in a single day. In this case the “AB” identifier may be omitted, and the date appended with a letter(s) incremented sequentially (i.e. “YYMMDDA.RAW”, “YYMMDDB.RAW”, “YYMMDDC.RAW” etc.).

Each acquisition program and data file is copied onto two portable disks; each disk is stored in a separate location. Original disks containing data acquisition programs are labeled “TCLT-PGM-ORIG-##”, backup disks containing data acquisition programs are labeled “TCLT-PGM-BKP-##”. Original disks containing data are labeled “TCLT-WAxxxx-ORIG-##”, backup disks containing data are labeled “TCLT-TWPxxxx-BKP-##” where:

"TC" indicates thermal conductivity

"LT" indicates LT apparatus
"PGM" indicates program disk
"ORIG" indicates original disk
"BKP" indicates a backup of a program or data disk
"TWPxxxx" is the Technical Workplan (TWP) number (TWP0000 if there is no TWP)
"##" is a sequential number of disks which otherwise have the same identifier.

Each portable disk must bear a label containing the following information:

- The TWP number and revision (TWP0000 if there is no governing TWP)
- The title, version number, and date copied of all program files on the disk.
- The filenames and of all data files on the disk.

Printouts of data must include appropriate units. Any data tabulated on a printout must be identified with descriptive headers.

4.3 Materials

A set of up to six reference specimens, of either Pyrex 7740 or high-purity fused quartz disks with nominal thickness' of 6 mm, 9 mm, 12 mm, 15 mm, 18 mm, and 21 mm, is used for instrument calibration and verification. If calibration results are consistently linear, then the reference set may be reduced to three disks with thicknesses that encompass the desired calibration range.

Dow Corning 340 Silicone Heat Sink Compound, or equivalent, may be used as a heat transfer medium to reduce the interfacial contact resistance (R_O) between the specimen's top and bottom surfaces and the adjacent heater plates, and to improve the reproducibility of the measured values of R_O.

5.0 PROCEDURES

All written records generated during the use of this TP, including logbook entries, are to be made using ink that will photocopy well.

All specimens will be identified, handled, shipped, and stored in accordance with QAIP 20-03, *Sample Control.* Calibrations of guarded heat-flow meters will be performed and documented (including labeling of the instruments) in compliance with the requirements of AP-12.1Q, *Control of Measuring and Test Equipment and Calibration Standards.* Any errors in the test control and data acquisition system or instructions discovered in following this TP shall be reported to the PI.

In this TP, messages or questions that appear on the computer monitor that require a response are indicated by "SCREEN: [message]". Responses to be entered are indicated by "ENTER: [response]". Striking the "Enter" key is indicated by "<Enter>".

5.1 Prerequisites

Verify and document as a permanent record that the following prerequisites collectively have been met:

(1) The most current version of this TP is being used.

(2) The correct documentation is present. This includes:

a) A Memorandum of Instruction (MI) from the Principal Investigator (PI) or designee. The MI shall include:

- Any necessary information regarding special specimen handling requirements, use of the moisture containment cell, etc.
- The specimens to be used in calibrations, verifications, or testing.
- The test temperatures to be used.

b) A Chain-of-Custody Form (QAIP 20-03, Appendix B) and Specimen Data Sheet (SDS) containing, at a minimum, the specimen identification, mass, and dimensions for each geologic specimen and each reference specimen.

c) Appropriate operating manual(s). Operating manuals are to be followed only when explicitly required by this TP; the TP otherwise incorporates the pertinent directions of the manuals.

(3) All instruments and equipment are clean.

(4) Each instrument and piece of equipment subject to calibration requirements (AP-12.1Q) has a current calibration documented by an attached label.

(5) Initial settings of the temperature controllers have been selected such that the guard temperature (T_g) is between the temperatures of the upper thermocouple (T_u) and the lower thermocouple (T_l) and such that ΔT is between 10 °C and 25 °C.

(7) All electrical cables and fluid hoses are properly connected (as specified in the instrument's operating manuals) and any necessary ancillary equipment (e.g., cooling system for the LT) is present and functional.

(8) The specimen is ready for testing (e.g., has the correct saturation state).

5.2 Step-by-Step Operating Procedure

5.2.1 Initial Conditions

Before the start of a set of a test, record the following information as a permanent record:

(1) Name and location of company performing the measurement.

(2) Identifier and revision of applicable TPs.

(3) Title and version number of test control and data acquisition routine.

(4) List of items to be used (including manufacturer, model, and serial number or other unique identifier).

(5) Name of person(s) performing the experiment.

(6) Date of test initiation.

(7) Acknowledgment that TP prerequisites have been met.

(8) Calibration date(s) of apparatus: Date of last calibration, verification, and check, date next calibration check is due, and signature of person verifying the calibration.

(9) Specimen identification (ID), material, as given on the Specimen Data Sheet. For geologic specimens, note the type of tuff (e.g., zeolitic, vitric, devitrified) if known. Provide the mass and thickness of each geologic specimen measured immediately before and after testing.

(10) Ambient laboratory conditions including room temperature and relative humidity.

(11) Initial settings, and any changes to settings, of the temperature controllers (described in operating manuals). Ramp rate of Eurotherm controller.

(12) Data, including units, or reference to any data recorded elsewhere (e.g., on computer printouts).

(13) Results and interim conclusions reached.

(14) Any and all unusual occurrences, deviations, nonconformances, and corrective actions.

(15) Modifications made to the technical requirements of the TP.

(16) Notation of any significant downtimes or delays.

(17) Name of the person making entry (printed, signed, and dated).

5.2.2 Measurement of Thermal Conductivity with the LT

5.2.2.1 Instrument Calibration

The LT is to be calibrated at least every six months when the instrument is in use. Each calibration must be verified by testing a reference specimen of a material not used in the

calibration. A label will be attached to the instrument indicating the temperature controller settings for which the current calibration is valid and when the next calibration is due. The LT must be re-calibrated when changes that may affect the calibration are made to the equipment (e.g., installation of a new HFT or new heater plates).

Measurement of a specimen in the moisture containment cell requires that the instrument be calibrated and verified with each reference specimen placed inside of a moisture containment cell. Heat sink compound may be used between the heater plates and the outer surfaces of the moisture containment cell.

Measurements on specimens with diameters less than 50.8 mm (2-in) require the use of similarly sized calibration and verification standards and moisture containment cells.

Check the calibration label on the instrument. If the calibration is current and no changes have been made to the temperature controller settings, proceed to Section 5.2.2.2. If the calibration is current, but the temperature controller settings have been changed, set the controllers to the values given on the calibration sticker and proceed to Section 5.2.2.2. If the calibration has expired, the following steps are to be performed:

(1) Obtain the 12 mm reference specimen (refer to the MI for specimen material) from the specimen storage facility to be used as the first.

(2) If the bell jar is positioned over the LT upper assembly, remove it.

(3) If not already on, turn on the power to the LT.

(4) Raise the upper heater of the LT by setting the toggle switch on the front panel to the "Stack Open" position.

(5) Swing the upper assembly to the left of the chamber.

(6) Wipe the reference specimen and the upper and lower heater plate faces free of any loose rock chips and dirt with a Kimwipe or similar material. A Kimwipe moistened with isopropyl alcohol may be used if traces of heat sink compound are present from a previous test.

(7) If instructed by the MI, spread a thin coating of heat sink compound on both faces of the specimen. Use only enough compound to completely cover the surfaces.

(8) Place the reference specimen within the Teflon ring on the lower heater plate in the test chamber. Rotate the specimen to ensure that it is seated properly.

(9) Swing the upper assembly to the right until it stops (it is now aligned with the chamber).

(10) Lower the assembly by setting the toggle switch to the "Stack Clamped" position.

(11) After lowering the assembly, check the cylinder air pressure (on the pressure gauge on the gas cylinder regulator). The pressure should be approximately 90 psi.

(12) Optional: Place the bell jar over the upper assembly.

(13) If not already on, turn on the cooling system and allow to equilibrate (approximately 2 hours).

(14) If not already on, turn on the computer, monitor, and printer.

(15) SCREEN: C:\TCA>

ENTER: TCA200LT <Enter>

(16) SCREEN: Automated Data Acquisition and Control for the

Thermatest TCA-200-LT Thermal Conductivity Instrument

Press <Enter> Key to Continue

(17) SCREEN: 1. CALIBRATION OF TCA-200-LT USING REFERENCE SAMPLE
2. MEASUREMENT OF CONDUCTIVITY OF UNKNOWN SAMPLE
3. VERIFICATION USING REFERENCE SAMPLE

ENTER 1, 2, or 3

ENTER: 1 <Enter>

(18) SCREEN: 1. CALIBRATION WITH PYREX ==> VERIFY WITH QUARTZ"
2. CALIBRATION WITH QUARTZ ==> VERIFY WITH PYREX"

ENTER 1 or 2

ENTER [1] or [2] <Enter>

(19) SCREEN: ENTER THE NAME OF THE FILE TO STORE RAW DATA IN

ENTER: [ABYYMMDD].RAW <Enter>

(where "ABYYMMDD" is assigned as described in Section 4.2.2.)

(20) SCREEN: ENTER UP TO TWENTY (20) CALIBRATION/TEST TEMPERATURES IN ºC. PRESS ENTER WHEN FINISHED WITH ENTRY SEQUENCE. AFTER ACQUIRING DATA AT THE CHOSEN

TEMPERATURES, THE LT WILL BE RETURNED TO THE FIRST TEMPERATURE ENTERED.

[The following values may be superseded by the MI]

SCREEN:	ENTER:
TEMP(1):	30<Enter>
TEMP(2):	50<Enter>
TEMP(3):	75<Enter>
TEMP(4):	100<Enter>
TEMP(5):	125<Enter>
TEMP(6):	150<Enter>
TEMP(7):	175<Enter>
TEMP(8):	200<Enter>
TEMP(9):	175<Enter>
TEMP(10):	150<Enter>
TEMP(11):	125<Enter>
TEMP(12):	100<Enter>
TEMP(13):	75<Enter>
TEMP(14):	50<Enter>
TEMP(15):	30<Enter>
TEMP(16):	<Enter>

(21) SCREEN: DO YOU WANT TO CHANGE THESE VALUES (Y/N)?

ENTER: N <Enter> [if values are those of Step (20)]
Y <Enter> [if values are not those intended in Step (20), and repeat Step (20)]

[In Step (20), given values are to be changed only if stipulated by the MI.]

(22) SCREEN: THE AVERAGING INTERVAL IS: 10 MINUTES
THE TEMPERATURE CRITERION IS: 2 DEGREES ºC
THE EQUILIBRIUM CRITERION IS: +/- 0.2 PERCENT
THE GUARD CRITERION IS: 0.25 DEGREES ºC
THE TIMEOUT LIMIT IS: 5 HOURS

DO YOU WANT TO CHANGE ANY OF THESE VALUES?

N <Enter> [if values are correct]
Y <Enter> [if MI values differ, then follow prompts]

(23) SCREEN: ENTER THE SAMPLE IDENTITY:

ENTER: [Type Specimen I.D.] <Enter>

SCREEN: ENTER THE SAMPLE THICKNESS IN MILLIMETERS:

ENTER: [Type length in mm] <Enter>

SCREEN: ADDITIONAL COMMENTS:

ENTER: [Type additional comments if desired] <Enter>

(24) The standard will be tested at each temperature entered in Step (20). When the test is completed, the program will set the controller back to the first temperature entered, and prompt:

SCREEN: DO YOU WISH TO CALIBRATE WITH ANOTHER STANDARD? (Y/N)

ENTER: Y<Enter> [if more standards are to be tested]

N<Enter> [if all standards have been tested]

(25) Repeat Steps (2), (4) and (5). Remove the specimen from the lower plate and use a Kimwipe or similar material to wipe off any loose debris. The Kimwipe may be moistened with isopropyl alcohol to remove any heat sink compound remaining if used. Return the specimen to the correct container.

(26) Repeat step 7, then place the next reference specimen within the Teflon ring on the lower heater plate in the test chamber. Rotate the specimen to ensure that it is seated properly. Repeat steps (9) through (12) and (23) through (25) for the remaining standards.

After all standards have been tested, a graph of R_S vs. $\Delta T/Q$ will be plotted to the screen and printed on the printer for each test temperature. Next, graphs of N vs. Temperature and R_0 vs. Temperature will be plotted and printed. Finally, the program will terminate and return to the DOS prompt.

(27) The calibration results have been stored in the file "TCACAL.DAT". Rename this file with a new filename:

SCREEN: C:\TCA>

ENTER: RENAME TCACAL.DAT [ABYYMMDD].LTC

(where "ABYYMMDD" is assigned as described in Section 4.2.2.)

(28) Make two backup copies of the file "ABYYMMDD.RAW" and “ABYYMMDD.LTC” each on a different portable disk labeled and stored as described in Section 4.2.2.

(29) Turn off cooling system.

(30) Turn off the LT, computer, printer, and monitor.

(31) Bring the test records and printouts to the PI or designee for review.

The PI or designee will check that the calibration was run at the correct temperatures, and that the results appear reasonable and indicate proper operation of the instrument. After the PI or designee has reviewed the results of the calibration, proceed to Section 5.2.2.2 to perform a verification of the calibration.

5.2.2.2 Calibration Verification

Each calibration of the LT must be verified by measuring the thermal conductivity of a high-purity reference specimen of a different material than that used for calibration. Then the measured values are compared with the accepted values (calculated from data published in NSRDS-NBS-8). After the PI or designee has determined that the calibration is valid, a label will be attached to the instrument certifying current calibration status.

Verifications will be run at least once a month when the instrument is in use. After each verification, a label will be attached to the instrument indicating when the next verification is due.

Check the label on the instrument to find the status of the verification. If the instrument is not due for a verification, proceed to Section 5.2.2.3 for measuring thermal conductivity of specimens. If a verification is required, the following steps are to be performed:

(1) Obtain the reference specimen (refer to the MI for specimen ID) from the specimen storage facility. Vespel SP1 may be used as a second verification.

(2) If the bell jar is positioned over the LT upper assembly, remove it.

(3) If not already on, turn on the power to the LT.

(4) Raise the upper heater of the LT by setting the toggle switch on the front panel to the "Stack Open" position.

(5) Swing the upper assembly to the left of the chamber.

(6) Wipe the reference specimen and the upper and lower heater plate faces free of any loose rock chips and dirt with a Kimwipe or similar material. A Kimwipe moistened with isopropyl alcohol may be used if traces of heat sink compound are present from a previous test.

(7) If instructed by the MI, spread a thin coating of heat sink compound on both faces of the specimen. Use only enough compound to completely cover the surfaces.

(8) Place the reference specimen within the Teflon ring on the lower heater plate in the test chamber. Rotate the specimen to ensure that it is seated properly.

(9) Swing the upper assembly to the right until it stops (it is now aligned with the chamber).

(10) Lower the assembly by setting the toggle switch to the "Stack Clamped" position.

(11) After lowering the assembly, check the cylinder air pressure (from the pressure gauge on the gas cylinder regulator). The pressure should be approximately 90 psi.

(12) Optional: Place the bell jar over the upper assembly.

(13) If not already on, turn on the cooling system and allow to equilibrate (approximately 2 hours).

(14) If not already on, turn on the computer, monitor, and printer.

(15) SCREEN: C:\TCA>

ENTER: TCA200LT <Enter>

(16) SCREEN: Automated Data Acquisition and Control for the

Thermatest TCA-200-LT Thermal Conductivity Instrument

Press <Enter> Key to Continue

(17) SCREEN: 1. CALIBRATION OF TCA-200-LT USING REFERENCE SAMPLE
2. MEASUREMENT OF CONDUCTIVITY OF UNKNOWN SAMPLE
3. VERIFICATION USING REFERENCE SAMPLE

ENTER 1, 2, or 3

ENTER: 3 <Enter>

(18) SCREEN: 1. CALIBRATION WITH PYREX ==> VERIFY WITH QUARTZ"
2. CALIBRATION WITH QUARTZ ==> VERIFY WITH PYREX"

ENTER 1 or 2

ENTER [1] or [2] <Enter>

(19) SCREEN: ENTER THE NAME OF THE FILE TO STORE RAW DATA IN

ENTER: [ABYYMMDD].RAW <Enter>

(where "ABYYMMDD" is assigned as described in Section 4.2.2.)

(20) SCREEN: ENTER UP TO TWENTY (20) CALIBRATION/TEST TEMPERATURES IN ºC. PRESS ENTER WHEN FINISHED WITH ENTRY SEQUENCE. AFTER ACQUIRING DATA AT THE CHOSEN TEMPERATURES, THE LT WILL BE RETURNED TO THE FIRST TEMPERATURE ENTERED.

[The following values may be superseded by the MI]

SCREEN:	ENTER:
TEMP(1):	30<Enter>
TEMP(2):	50<Enter>
TEMP(3):	75<Enter>
TEMP(4):	100<Enter>
TEMP(5):	125<Enter>
TEMP(6):	150<Enter>
TEMP(7):	175<Enter>
TEMP(8):	200<Enter>
TEMP(9):	175<Enter>
TEMP(10):	150<Enter>
TEMP(11):	125<Enter>
TEMP(12):	100<Enter>
TEMP(13):	75<Enter>
TEMP(14):	50<Enter>
TEMP(15):	30<Enter>
TEMP(16):	<Enter>

(21) SCREEN: DO YOU WANT TO CHANGE THESE VALUES (Y/N)?

ENTER: N <Enter> [if values are those of Step (20)]
Y <Enter> [if values are not those intended of Step (20), and repeat Step (20)]

[In Step (20), given values are to be changed only if stipulated by the MI.]

(22) SCREEN: THE AVERAGING INTERVAL IS: 10 MINUTES
THE TEMPERATURE CRITERION IS: 2 DEGREES ºC
THE EQUILIBRIUM CRITERION IS: +/- 0.2 PERCENT
THE GUARD CRITERION IS: 0.25 DEGREES ºC
THE TIMEOUT LIMIT IS : 5 HOURS

DO YOU WANT TO CHANGE ANY OF THESE VALUES?

N <Enter> [if values are correct]
Y <Enter> [if MI values differ, then follow prompts]

(23) SCREEN: ENTER NAME OF CALIBRATION FILE:

ENTER: [Type name of calibration file to be verified] <Enter>

(24) SCREEN: ENTER THE SAMPLE IDENTITY:

ENTER: [Type Specimen I.D.] <Enter>

SCREEN: ENTER THE SAMPLE THICKNESS IN MILLIMETERS:

ENTER: [Type length in mm] <Enter>

SCREEN: ADDITIONAL COMMENTS:

ENTER: [Type additional comments if desired] <Enter>

(25) The specimen will be tested at each temperature entered in Step (20). For each temperature, the measured specimen conductivity and the calculated value of the conductivity of the reference material at that temperature will be printed. In addition, the difference between the measured and calculated value in W/mK and in percent will be printed. When the test is completed, the program will set the controller back to the first temperature entered. Next the program will plot to the screen, then print a graph of Conductivity vs. Temperature. Finally, the program will terminate and return to DOS.

(26) Repeat Steps (2), (4) and (5). Remove the specimen from the lower plate and use a Kimwipe or similar material to wipe off any loose debris. The Kimwipe may be moistened with isopropyl alcohol to remove any heat sink compound if used. Return the specimen to the correct container.

(27) Turn off cooling system.

(28) Make two backup copies of the file "ABYYMMDD.RAW" each on a different portable disk labeled and stored as described in Section 4.2.2.

(29) Turn off the LT, computer, printer, and monitor.

(30) Bring the test records and printouts to the PI or designee for review.

The PI or designee will compare the difference between the measured and accepted thermal conductivity values of the specimen as they appear on the printout. At each temperature, the measured value must be within ±0.2 W/mK of

the accepted value. If the difference is greater than ±0.2 W/mK, a new calibration must be performed as described in Section 5.2.2.1, and any data taken since the last successful verification must be evaluated for acceptability by the PI.

A label will be attached to the instrument certifying its calibration status as current. The label shall include the serial number or other unique identifier of the instrument, the signature(s) of the person(s) who performed the verification and the date when the next verification is due. After this label is attached, proceed to Section 5.2.2.3 for measuring thermal conductivity of specimens.

5.2.2.3 Measurement of Thermal Conductivity of Specimens

(1) Obtain the test specimen (refer to the MI for specimen ID) from the specimen storage facility.

(2) If the bell jar is positioned over the LT upper assembly, remove it.

(3) If not already on, turn on the power to the LT.

(4) Raise the upper heater of the LT by setting the toggle switch on the front panel to the "Stack Open" position.

(5) Swing the upper assembly to the left of the chamber.

(6) Wipe the test specimen and the upper and lower heater plate faces free of any loose rock chips and dirt with a Kimwipe or similar material. Measure and record the pretest specimen thickness and mass on the SDS. A Kimwipe moistened with isopropyl alcohol may be used if traces of heat sink compound are present from a previous test.

(7) If instructed by the MI, spread a thin coating of heat sink compound on both faces of the specimen. Use only enough compound to completely cover the surfaces.

(8) Place the specimen within the Teflon ring on the lower heater plate in the test chamber. Rotate the specimen to ensure that it is seated properly.

(9) Swing the upper assembly to the right until it stops (it is now aligned with the chamber).

(10) Lower the assembly by setting the toggle switch to the "Stack Clamped" position.

(11) After lowering the assembly, check the cylinder air pressure (from the pressure gauge on the gas cylinder regulator). The pressure should be approximately 90 psi.

(12) Optional: Place the bell jar over the upper assembly.

(13) If not already on, turn on the cooling system and allow to equilibrate (approximately 2 hours).

(14) If not already on, turn on the computer, monitor, and printer.

(15) SCREEN: C:\TCA>

ENTER: TCA200LT <Enter>

(16) SCREEN: Automated Data Acquisition and Control for the

Thermatest TCA-200-LT Thermal Conductivity Instrument

Press <Enter> Key to Continue

(17) SCREEN: 1. CALIBRATION OF TCA-200-LT USING REFERENCE SAMPLE
2. MEASUREMENT OF CONDUCTIVITY OF UNKNOWN SAMPLE
3. VERIFICATION USING REFERENCE SAMPLE

ENTER 1, 2, OR 3

ENTER: 2 <Enter>

(18) SCREEN: ENTER THE NAME OF THE FILE TO STORE RAW DATA IN

ENTER: [ABYYMMDD].RAW <Enter>

(where "ABYYMMDD" is assigned as described in Section 4.2.2.)

(19) SCREEN: ENTER UP TO TWENTY (20) CALIBRATION/TEST TEMPERATURES IN ºC. PRESS ENTER WHEN FINISHED WITH ENTRY SEQUENCE. AFTER ACQUIRING DATA AT THE CHOSEN TEMPERATURES, THE LT WILL BE RETURNED TO THE FIRST TEMPERATURE ENTERED.

[The following values may be superseded by the MI]

SCREEN:	ENTER:
TEMP(1):	30<Enter>
TEMP(2):	50<Enter>
TEMP(3):	75<Enter>
TEMP(4):	100<Enter>
TEMP(5):	125<Enter>

SCREEN:	ENTER:
TEMP(6):	150<Enter>
TEMP(7):	175<Enter>
TEMP(8):	200<Enter>
TEMP(9):	175<Enter>
TEMP(10):	150<Enter>
TEMP(11):	125<Enter>
TEMP(12):	100<Enter>
TEMP(13):	75<Enter>
TEMP(14):	50<Enter>
TEMP(15):	30<Enter>
TEMP(16):	<Enter>

(20) SCREEN: DO YOU WANT TO CHANGE THESE VALUES (Y/N)?

ENTER: N <Enter> [if values are those of Step (19)]
Y <Enter> [if values are not those intended of Step (19), and repeat Step (19)]

[In Step (19), given values are to be changed only if stipulated by the MI.]

(21) SCREEN: THE AVERAGING INTERVAL IS: 10 MINUTES
THE TEMPERATURE CRITERION IS: 2 DEGREES ºC
THE EQUILIBRIUM CRITERION IS: +/- 0.2 PERCENT
THE GUARD CRITERION IS: 0.25 DEGREES ºC
THE TIMEOUT LIMIT IS: 5 HOURS

DO YOU WANT TO CHANGE ANY OF THESE VALUES?

N <Enter> [if values are correct]
Y <Enter> [if MI values differ, then follow prompts]

(22) SCREEN: ENTER NAME OF CALIBRATION FILE:

ENTER: [Type name of calibration file to be verified] <Enter>

(23) SCREEN: ENTER THE SAMPLE IDENTITY:

ENTER: [Type Specimen I.D.] <Enter>

SCREEN: ENTER THE SAMPLE THICKNESS IN MILLIMETERS:

ENTER: [Type length in mm] <Enter>

SCREEN: ADDITIONAL COMMENTS:

ENTER: [Type additional comments if desired] <Enter>

(24) The specimen will be tested at each temperature entered in Step (19). When the test is completed, the program will set the controller back to the first temperature entered. Next the program will plot to the screen, then print a graph of sample Conductivity vs. Temperature on the printer. Once the graph is printed, the program will terminate and return to DOS.

(25) Repeat Steps (2), (4) and (5). Remove the specimen from the lower plate and use a Kimwipe or similar material to wipe off any loose debris. The Kimwipe may be moistened with isopropyl alcohol to remove any heat sink compound if used. Measure and record post test specimen thickness and mass on the SDS. The difference between the pretest and posttest masses may be affected by incomplete removal of the heatsink compound. Return the specimen to the correct container.

(26) Turn off cooling system.

(27) Make two backup copies of the file "ABYYMMDD.RAW" each on a different portable disk labeled and stored as described in Section 4.2.2.

(28) Turn off the LT, computer, printer, and monitor.

(29) Bring the test records and printouts to the PI or designee for review. The PI or designee will check that the test was conducted at the correct temperatures, that the results were within the calibrated range of the instrument, and that the results appear reasonable.

5.3 Postrequisites

Verify that:

(1) Each data file has been copied onto two portable disks. Each portable disk is labeled and stored as specified in Section 4.2.2.

(2) All equipment has been turned off.

(3) Each specimen has been returned to the correct container and handled and stored in accordance with the requirements of QAIP 20-03, *Sample Control.*

(4) Each test record entry is complete, signed, and dated.

5.4 Hold Points

The following hold points have been identified in this TP:

1. Section 5.2.2.1 (31): The LT calibration is completed.

2. Section 5.2.2.2 (30): The LT verification is completed.

3. Section 5.2.2.2 (30): The results of the LT calibration verification are not in the acceptable range or the measured resistance of the specimen is outside of the resistance range over which the instrument was calibrated.

4. Section 5.2.3.3 (29): The conductivity test with the LT is completed.

5. Section 5.2.3.3 (29): The measured resistance of the specimen is outside of the resistance range over which the instrument was calibrated.

These hold points require notification of, and instructions from, the PI or designee before continuing with use of the procedure.

Any nonconformances or deviations must be reported to the Principal Investigator as soon as possible. Deviations, deficiencies, and corrective actions must be determined and documented in accordance with AP-15.2Q, *Control of Nonconformances* and AP-16.1Q, *Management of Conditions Adverse to Quality.*

All reviews by the PI or designee will be documented as a permanent record.

6.0 Safety

There should be no safety hazards other than the normal hazards (e.g., hot heating plates) of the equipment; all equipment should be operated in accordance with the safety requirements of the facility where the work is being performed.

7.0 QA RECORDS

QA records, and any corrections or changes thereto, generated as a result of implementing this procedure will be prepared and submitted as inclusionary QA records (QA:QA) by the Principal Investigator in accordance with AP-17.1Q, *Record Source Responsibilities for Inclusionary Records.* These records include:

- Proficiency Training Records (Section 2.0)
- Prerequisite Documentation (MI, Chain-of-Custody Forms as per Section 5.1 Step 2)
- Initial Conditions (Section 5.2.1, Items 1 - 17)
- Test or Calibration Records and PI review (Section 5.2.2.1 Step 34, Section 5.2.2.2 Step 31, and Section 5.2.2.3 Step 31.)

8.0 REFERENCES

American Society for Testing and Materials (ASTM) E177-86, "Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods," April 1986.

American Society for Testing and Materials (ASTM) F433-77 (Reapproved 1987), "Standard Practice for Evaluating Thermal Conductivity of Gasket Materials," May 1977.

Powell, R.W., Ho, C.Y., and Liley, P.E., 1966, "Thermal Conductivity of Selected Materials," NSRDS-NBS-8, National Bureau of Standards Reference Data Series.

"Operating Manual for the C-Matic Heat Flow Meter Thermal Conductance Tester Model TCHM-LT," Holometrix, Inc.

AP-12.1Q, *Control of Measuring and Test Equipment and Calibration Standards*

AP-15.2Q, *Control of Nonconformances*

AP-16.1Q, *Management of Conditions Adverse to Quality*

AP-17.1Q, *Record Source Responsibilities for Inclusionary Records*

QAIP 20-01, *Technical Procedures*

QAIP 20-03, *Sample Control*

TP-200, *Inspection of Samples Used in Thermal Properties Measurements*

TP-215, *Calibration of Lawson Board Systems*

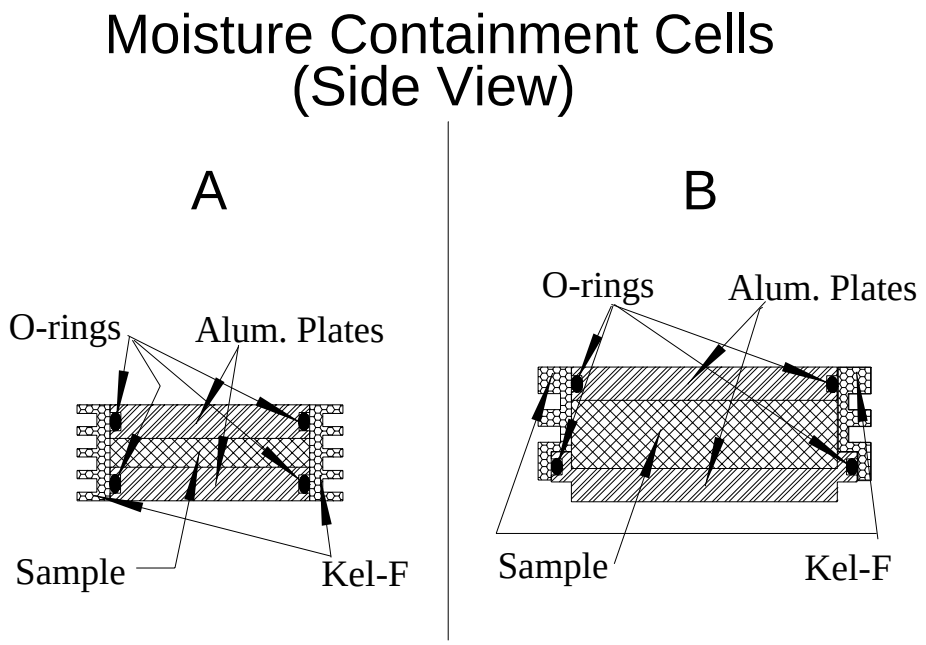

Figure 1A: 1.5" Diameter Moisture Containment Cell; Figure 1B: 2.0" Diameter Moisture Containment Cell

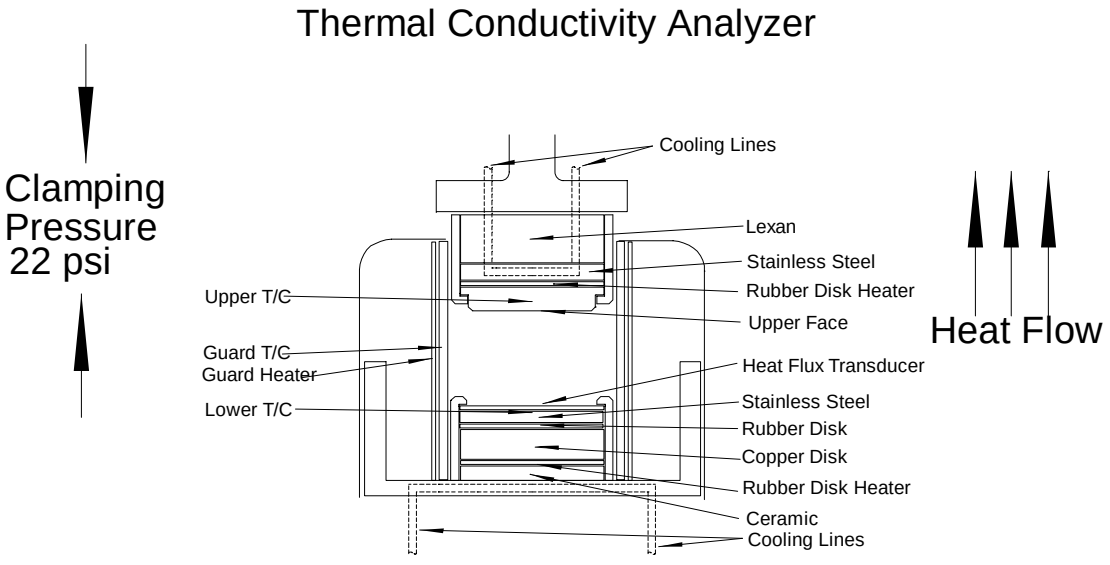

Figure 2: C-Matic LT Guarded Heat Flow Meter Diagram